PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of:		) Paper No. 6
APPLICANTS:	GEORGE A. HUFF JR. LARRY W. KRUSE, OZIE S. OWEN, MONICA R. REGALBUTO & WILLIAM A. GONG	)
SERIAL NO:	09/779,284	) Group Art Unit:) 1764
FILED:	February 8, 2001	) Examiner:) Walter D. Griffin
FOR:	HYDROTREATING OF COMPONENTS FOR REFINERY BLENDING OF TRANSPORTATION FUELS	) Attorney Docket) No.: 37,248-02

AFFIDAVIT UNDER 37 CFR § 1.131
of Dr. GEORGE A. HUFF Jr.

Assistant Commissioner for Patents
Washington, DC 20231

CERTIFICATE OF MAILING

I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to: Commissioner of Patents and Trademarks, Washington, D.C. 20231

on Sept. 24 2002. By Carol M. Neth

(Date) (Signature)

CAROL M. NETH

(Typed or Printed Name f Person Signing Certificate)

1. I, GEORGEA. HUFF Jr., being duly sworn, depose and say:

2. That I reside at 823 Morven Court, Naperville, Illinois 60563.

3. In May 1984 I received a Bachelor of Science degree in Chemical Engineering from the University of Utah, Salt Lake, Utah, USA. I received a Doctor of Philosophy Chemical Engineering in 1982 from the Massachusetts Institute of Technology, Cambridge, Massachusetts, USA.

4. 1982 to 1984, I held the position of Assistant Professor of Chemical Engineering at M.I.T.

5. 1984 to 1986, I was a Research Engineer working in the Hydrotreating Group of the Catalysis Department for Shell Development at Westhollow Technical Center, Houston, Texas, USA.

6. From 1986 to the present, I have been employed by Amoco Chemical Company, now BP Amoco Chemical Company, a corporation of the State of Delaware, and have the position of Senior Research Associate. Among the professional honors which have been conferred on me are memberships in the North American Catalysis Society and the American Chemical Society.

7. I am one inventor of the claimed subject matter of the above identified patent application.

8. I have read U.S. Patent No 6,217,748 in the name of Hatanaka et al., and entitled PROCESS FOR HYDRODESULFURIZATION OF DIESEL GAS OIL.

9. Prior to April of 2000, our invention as described and claimed in the subject application was completed in the United States, as evidenced by the following Exhibits:

a Pages 1 and 2 of memorandum titled PRODUCTION OF LOW SULFUR DIESEL (25 PPM AND 150 PPM) by S. G. McDaniel and M. A. Jandick for Amoco Petroleum Products, Naperville, Illinois, identified as EXHIBIT A, illustrates the key points of our selective hydrogenation of high-boiling hydrogenation feedstock whereby the incorporation of hydrogen into hydrocarbon compounds, sulfur-containing organic compounds, and/or nitrogen-containing organic compounds assists by hydrogenation removal of sulfur and/or nitrogen from components for refinery blending of transportation fuels.

b Table 1 : Properties of Feed, identified as EXHIBIT B.

c Table 2 : Properties of Catalyst, identified as EXHIBIT C.

d Table 4 : Properties for < 30 ppm Composite Sulfur Product, identified as EXHIBIT D.

10. As EXHIBIT A, illustrates, a hydrotreated desulfurized diesel having 375 ppm sulfur was used as the feed to the hydrotreating pilot plant to make products having reduced sulfur levels of about 150 ppm sulfur and less than 30 ppm sulfur. The feed was designated as LS-98. As stated on page 2, "The hydrotreated feed was difficult to desulfurize since 80 percent of the sulfur compounds boiled above 600° F. The majority of these sulfur compounds are dibenzothiophenes and substituted dibenzothiophenes. We had to run the unit with fresh catalyst at 680° F to achieve the 25 ppm product sulfur level."

11. Properties of the feed are summarized in Table 1, identified as EXHIBIT B.

12. The fresh catalyst used is one of the more active CoMo catalysts on the market for desulfurization of petroleum distillates. Selected properties of the catalyst are summarized in Table 2, identified as EXHIBIT C.

13. During the run, the majority of the sulfur samples were tested using analytical methods SPPM1640 and GCSBP2360. The

results were confirmed periodically by analyzing the same sample by method SXRF12740. Properties for <30 ppm Composite Sulfur Product are summarized in Table 4, identified as EXHIBIT D. These results demonstrate the effectiveness of our procedure in reducing the sulfur and nitrogen content of LS-98-150-A600, which originally contained 350 ? or 375 ppmw sulfur and 89 ppmw nitrogen.

15. Copies of the above referenced memorandum pages are attached as Exhibits. The Exhibits are a true copy, except that the dates thereof and unrelated subject matter have been blanked-out, but all the dates are prior to August 1999.

AND FURTHER AFFIANT SAYETH NOT.

GEORGE A. HUFF Jr.

STATE OF ILLINOIS)
) SS.
COUNTY OF DUPAGE)

Sworn to and subscribed before me, a Notary Public, by said GEORGE A. HUFF Jr., on this 19th day of September 2002.

(SEAL)

Notary Public

"OFFICIAL SEAL"
JANE L. COOPER
Notary Public, State of Illinois
My Commission Expires 06/01/04

MEMORANDUM

Amoco Petroleum Products
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PRODUCTION OF LOW SULFUR DIESEL (25 PPM AND 150 PPM)

INTRODUCTION

testing. A desulfurized diesel from Whiting (LS-98) was used as the feed to the pilot plant to make seven barrels of 150 ppm sulfur diesel and seven barrels with less than 30 ppm sulfur. This memorandum documents feed and product analyses along with associated pilot plant processing conditions.

OPERATION

Feed

Hydrotreated HMD (LS-98 diesel) from the Whiting DDU was used as the feed to AU-125. Since the feed is already hydrotreated, the remaining 350 ppm sulfur is heavy: approximately 80% of the sulfur boils above 600°F. The properties for the feed as analyzed by the Amoco Research Center are listed in Table I.

Catalyst

We loaded 664 grams (950 cc) of fresh
catalyst and 150 cc silicon carbide into the reactor. This catalyst is currently used in the distillate desulfurization unit at the Amoco Yorktown refinery and is one of the more active CoMo catalysts on the market for desulfurization. The catalyst properties as tested at the Amoco Research Center are listed in Table II.

Conditions

EOR 740°F). The conditions for the pilot plant were as follows: pressure 500 to 550 psig, pure H_2, temperature 600°F-680°F. Sulfur samples were taken every day or two to monitor the quality of the product and detect upsets in the unit.

PRODUCTS/RESULTS

Two product composites were produced for this project, a 150 ppm sulfur diesel and a maximum 30 ppm sulfur diesel. The actual sulfur levels were Composite #1--151 ppm and Composite #2--25 ppm, respectively. The product properties for both sulfur levels as analyzed at the Amoco Research Center in Naperville are listed in Tables III and IV, respectively. Barrels 2, 3, 4, 5, 7, 8, and 9 were blended to make Composite #1. Barrels 10, 11, 12, 13, 14, 16, 17, 18, and 19 were blended to make Composite #2. The other barrels were used to flush the blending tank before each operation.

The main parameter tracked during the run was product sulfur concentration in ppm. The majority of the sulfur samples were tested using analytical method SPPM1640. The results were confirmed periodically by sending the same sample in for SXRF1240. A plot of sulfur concentration per period is shown in Figure 1.

SUMMARY

The AU-125 pilot run was executed efficiently, and the composites produced were on target. The hydrotreated feed was difficult to desulfurize since 80% of the sulfur compounds boiled above 600°F. The majority of these compounds are dibenzothiophenes and substituted dibenzothiophenes. We had to run the unit with fresh catalyst at 680°F to achieve the 25 ppm product sulfur level. Most of the Amoco distillate desulfurization units (DDUs)

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Attachments

Keywords: Hydrotreating, Diesel, Low Sulfur, Pilot Plant, DC-130

TABLE I
PROPERTIES OF FEED (LS-98 DIESEL FROM WHITING)

Physical Properties	Result	Test Code	Volume Percent	IBP Distillation (FILD86DI :T)	Sulfur by Boiling Pt. (GCSBP 2360)
Sulfur (ppm)	375	S PPM 1640	IBP 0.5%	270	440
Nitrogen (ppm)	89	N PPM 1560	1.0 %	292	453
Aromatic carbon (wt%)	16.5	NMRC 6831	5.0 %	355	522
API Gravity	34.66	FLAPIG 9710	10.0 %	384	554
Sp. gravity	0.8516	FLAPIG 9710	20.0 %	429	615
Cetane Index (4 point)	46.946	FILCETIND4/ 1	30.0 %	457	633
Carbon (wt%)	86.96	CHHIGH 1450	40.0 %	490	663
Hydrogen (wt%)	13.11	CHHIGH 1450	50.0 %	523	667
			60.0 %	549	676
			70.0 %	575	693
			80.0 %	605	705
			90.0 %	636	727
			95.0 %	663	746
			99.0 %	714	825
			FBP 99.5 %	733	855

TABLE II
PROPERTIES OF CATALYST

			Metals Analysis for CHD-1682			
Physical Properties	Result	Test Code	Test Code: ICP 1340		Test Code: XRF 1040	
BET Surface Area m2/g	236	5170 SA	Na ppm		Na Wt%	-
Cum. Desorption cc/g	0.48	5170 SA	Mg ppm		Mg Wt%	-
Avg. Pore volume A	29	5170 SA	Al ppm	3E	Al Wt%	
Crush Strength #/mm	3.60	-	Si ppm		Si Wt%	
CBD g/cc	0.71	-	P ppm		P Wt%	
			K ppm	<	K Wt%	
			Ca ppm		Ca Wt%	
			Ti ppm		Ti Wt%	
			V ppm		V Wt%	
			Cr ppm		Mn Wt%	
			Mn ppm		Fe Wt%	
			Fe ppm		Co Wt%	
			Co ppm		Ni Wt%	
			Ni ppm		Zn Wt%	
			Zn ppm		Mo Wt%	
			Mo ppm		Sn Wt%	
			Pb ppm		Sb Wt%	
					Ba Wt%	
					La Wt%	
					Ce Wt%	
					Nd Wt%	

TABLE III

PROPERTIES OF 150 PPM COMPOSITE SULFUR PRODUCT

Physical Properties	Result	Test Code	Volume Percent	IBP Distillation (FILD86DIST)	Sulfur by Boiling Pt. (GCSBP 2360)
Sulfur (ppm)	151	S PPM 1640	IBP 0.5 %	333	35
Nitrogen (ppm)	33	N PPM 1560	1.0 %	-	135
Aromatic carbon (wt%)	15.6	NMRC 6831	5.0 %	394	522
API Gravity	35	FLAPIG 9710	10.0 %	421	572
Sp. gravity	0.8498	FLAPIG 9710	20.0 %	448	645
Cetane Index (4 point)	46.836	FILCETIND4/1	30.0 %	473	663
Carbon (wt%)	86.45	CHHIGH 1450	40.0 %	494	668
Hydrogen (wt%)	13.07	CHHIGH 1450	50.0 %	512	676
			60.0 %	532	687
			70.0 %	552	695
			80.0 %	574	708
			90.0 %	603	733
			95.0 %	630	757
			99.0 %	-	851
			FBP 99.5 %	643	893

TABLE IV

PROPERTIES FOR < 30 PPM COMPOSITE SULFUR PRODUCT

Physical Properties	Result	Test Code	Volume Percent	IBP Distillation (FILD86DIST)	Sulfur by Boiling Point (GCSBP 2360)
Sulfur (ppm)	25	S PPM 1640	IBP 0.5 %	349	20
Nitrogen (ppm)	17	N PPM 1560	1.0 %	-	46
Aromatic carbon (wt%)	14.8	NMRC 6831	5.0 %	408	238
API Gravity	35.3	FLAPIG 9710	10.0 %	428	640
Sp. gravity	0.8485	FLAPIG 9710	20.0 %	453	661
Cetane Index (4 point)	47.813	FILCETIND4/1	30.0 %	474	668
Carbon (wt%)	86.72	CHHIGH 1450	40.0 %	495	673
Hydrogen (wt%)	13.12	CHHIGH 1450	50.0 %	514	688
			60.0 %	532	695
			70.0 %	552	706
			80.0 %	573	719
			90.0 %	600	741
			95.0 %	625	767
			99.0 %	-	857
			FBP 99.5 %	647	910